IX.

ON THE PROCESS OF REVERSE FILTERING AND ITS APPLICATION TO LARGE MASSES OF MATERIAL.

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By reverse filtering is meant a process of filtration in which the liquid to be filtered is drawn upwards instead of flowing downwards in the usual way. Such a system is often used in the arts, as when a porous septum is attached to the mouth of a suction pipe; or as in the small portable filters so useful to travellers, by which clear water may be sucked up from a muddy pool or turbid stream. These last suggested the application of the same principle in chemical analysis to the treatment of those precipitates which are usually weighed on a dried filter. In such cases, it is of course essential that the weight of the paper disk used as a filter should remain invariable; and this constancy can be best secured by making the disk as small as possible. If the filter is large, it is impossible to have any confident assurance of the constancy of its weight, however great the care that may be taken to secure a similarity of hygrometric conditions at the two weighings; and hence it has not hitherto been practicable to determine on a dried filter the weight of any considerable quantity of a precipitate with accuracy. But, in the process of reverse filtering, we can both wash and collect very large masses of precipitates with a filter not more than an inch in diameter; and if, before drying, these little disks of paper are soaked in dilute hydrochloric acid, and afterwards thoroughly washed in water, their weight remains practically invariable. Indeed, it is not necessary to enclose the filter in a weighing tube, or to pay any special regard to its hygrometric conditions other than to keep the usual drying materials in the balance case. The only liability to alteration of weight would arise from the dissolving of soluble material in the paper, and this may be wholly prevented by previously washing the disks as just described.

In the early part of 1873, having occasion to determine large quan-

tities of sulphide of antimony, we in the first place employed the porous filtering cones described by Prof. C. E. Munroe; * but we found these both too limited in capacity, and too susceptible to hygroscopic influences, to give the degree of accuracy we required. We were therefore led to devise the following apparatus, which Figures 1 and 2 will help us to describe. The most essential part of this apparatus is the platinum "rose" represented by Fig. 1. This is cemented by sealing-wax to the end of a glass tube, and to its perforated base the small filters are applied. The glass tube is so cemented into the neck of the "rose" that the end may reach quite down to the perforated plate, and thus draw up all the liquid which collects in the hemispherical cup. Moreover, the perforated plate has an unbroken rim about $\frac{1}{16}$ of an inch wide around the edge, which is sufficient to prevent any solid particles from creeping by the edges of the paper disk. The success and rapidity of the filtering depend on the proper construction of the perforated disk, and we obtained the best results only after several trials. The holes should be smoothly perforated about $\frac{1}{50}$ of an inch in diameter, and as numerous as possible, leaving the unbroken rim described above. After the perforations are made, the face of the plate should be ground perfectly smooth. We use two sizes of these "roses," in one of which the hemisphere is $\frac{5}{16}$ of an inch, and of the other $1\frac{1}{4}$ inches in diameter; but the smaller is the more useful, and is sufficiently large for all ordinary purposes. A disk of washed Swedish filtering paper, $\frac{5}{16}$ of an inch in diameter, weighs only about 20 milligrammes; and, to give an idea of the rapidity of the filtration, it is sufficient to say that, under a pressure of 50 centimetres of mercury, these filters will pass from 20 to 30 litres of clear water in an hour. We have been greatly indebted to Messrs. Johnson, Matthey, & Co., of Hatton Garden, London, for the care they have taken in the construction of these "roses;" and they can be procured of them through the mail. The cost of the smaller size is fifteen shillings sterling. The construction of the rest of the apparatus is made clear by Fig. 2. One of the stems of a glass three-way tube is clamped to an arm which can be raised or lowered on the vertical bar of an elevating stand by a rack and pinion movement, which ought, however, to be so loose, that the arm can be pushed suddenly up when necessary. By its second stem, the three-way tube is connected

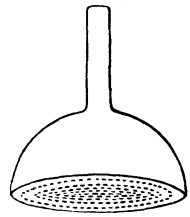

Fig. 1.

* American Journal of Science, May, 1871.

by a rubber hose with a large glass bottle, in which a partial vacuum is maintained by a Bunsen pump, but this connection can be closed by

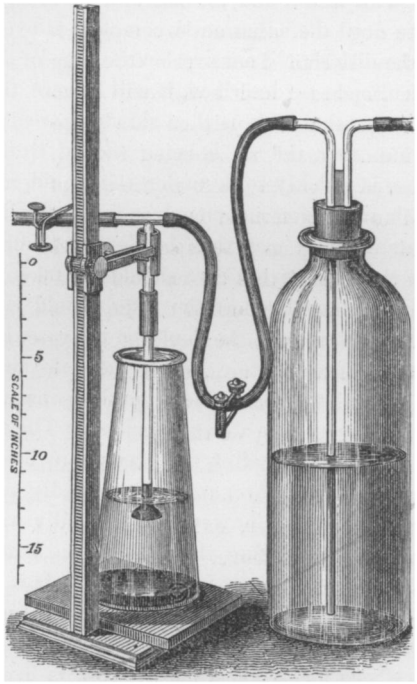

Fig. 2.

a compression cock. The third stem, which makes a connection with the atmosphere, is closed by a rubber connector and nipper tap, and serves to suddenly relieve the pressure in case the filter slips or breaks. From the first and vertical stem of the three-way tube below the clamp is suspended the glass tube, having the rose at its end. The suspension consists of a rubber connector, so long that it can be bent double and the rose inverted, and so stout that when thus bent the connection with the bottle is completely closed. When inverted, the tube of the rose rests against the thumbscrew of the pinion, and is thus confined.

In order that we may make clear the mode of using the apparatus, let us assume that five grammes of antimonious sulphide have been precipitated in a glass beaker, holding two litres of liquid, and that the precipitate has settled, — as it will after boiling, — leaving the supernatant liquid perfectly clear. A partial vacuum having been formed

in the bottle, and the beaker having been placed on the stand, we begin by attaching the paper disk to the base of the rose, moistening it for the purpose with water, and forcing it with the thumb against the perforated plate until the adhesion is complete at every point. It is important that the disk should not overlap the edge of the plate; and, if the plate has been made as described, it will adhere tightly without so doing. The compression cock is then slowly opened; and, as soon as the sound indicates that the air is being sucked through the filter, the base of the rose is quickly sunk under the liquid, and constantly lowered by the rack and pinion movement, as fast as the liquid is drawn off. The supernatant liquid may thus be decanted until the rose is close down to the precipitate; but care should be taken at this stage not to push the process too far, lest the filter should become clogged by the adhesion of solid particles, as would be the case, if it comes too near the level of the precipitate. When the safe level has been reached, the arm of the stand is suddenly pushed upwards, while the rose is inverted and supported as above described. The beaker is now filled up with hot water; and, after the precipitate has subsided, the wash water is drawn off as before, and so repeatedly as often as may be necessary. It is important, however, to carefully watch the filter, and every time before immersing the rose to see that the paper is moist and firmly adhering to the perforated plate. It is further essential that the suction should not be for a moment interrupted while the filter is immersed. This condition is secured by the simple rubber joint we have described; for, while when the rose is inverted the connection with the bottle is closed, the moment it is turned down the connection is opened, and the filter begins to draw. If, however, by any accident the filter should slip, the operator when on his guard can avoid loss of material by quickly opening the nipper tap, and relieving the pressure until the rose can be withdrawn and washed out. The filter can then be fished out with a glass rod, washed off and replaced. A good strong filter will bear quite rough treatment; and, if in the process it becomes clogged, it can be taken off when the rose is inverted, and the adhering precipitate washed back into the beaker. When replaced, the paper thus cleansed often filters as rapidly as before. If, as is sometimes the case, the paper becomes hopelessly clogged, no great loss of accuracy is suffered by using a second or even a third filter. Of course, they must all be dried, and weighed either with the precipitate or apart, as most convenient.

The precipitate, having been thus washed, is next to be transferred to the crucible in which it is to be weighed; and, to hold five grammes

of precipitated antimonious sulphide, we shall require a crucible having a capacity of 250 cubic centimetres. As much of the water as can readily be decanted from the precipitate is first poured into the crucible and drawn off with the filter, and then the precipitate is washed in with as little additional water as possible. Now the filter is plunged into the semi-fluid mass, and must not afterwards be removed until the process is completed. The rose ought not even to be raised however slightly, although additional material may be poured in around it. As the mass contracts in the crucible, the filter must be made to follow, always keeping it immersed; and during this time the precipitate which has collected around the platinum rose may be washed down by a stream from a wash bottle. In this way the greater part of the water can be removed, leaving the precipitate nearly as compact as it is left on a common filter, when dried by Bunsen's pump. When the precipitate is in this condition, the pressure is relieved by opening the nipper tap, and the rose raised, which leaves the filter behind. If any of the precipitate has clung to the platinum, this must now be washed into the crucible with a few drops of water, the rose and tube having first been detached from the connector for the purpose. It only now remains to dry the precipitate with the little filter, and weigh it. If it is important to dry the precipitate at a temperature above 150° C., or even to ignite it, the mass should first be thoroughly dried at 100°. The little disk of paper can then be removed and weighed separately, while the rest of the mass is heated to a higher temperature. The amount of material which remains adhering to the paper under these circumstances is exceedingly small, not usually exceeding a few milligrammes; and allowance can be made for it in the final result, without sensible error.

Like other analytical processes, this method has its limits; and any attempt to extend it beyond the sphere of its usefulness will lead to unsatisfactory results. It is of no use for filtering turbid liquids, since the small filters are rapidly clogged, and the process becomes proportionally slow. Whenever, however, a precipitate settles clearly, this method enables us to wash, collect, and weigh very large quantities of precipitates in a very short time with wonderful accuracy. We have used it chiefly to determine sulphide of antimony and chloride bromide or iodide of silver; and the results of the following analyses, extracted from our forthcoming paper on the Atomic Weights of Antimony, will show how great accuracy can be attained with it: —

SYNTHESIS OF SULPHIDE OF ANTIMONY.

Weight in Grammes of Sb taken.	Weight of $Sb_2 S_3$ dried at 180° C.	Per cent of S in same.
2.1439	3.0025	28.58
2.3417	3.2792	28.59
2.2182	3.1061	28.59

ANALYSIS OF ANTIMONIOUS CHLORIDE.

Weight in Grammes of $Sb Cl_3$ taken.	Weight of AgCl obtained.	Per cent of Chlorine
2.0300	3.8282	46.652
1.3686	2.5813	46.659
1.8638	3.5146	46.651

ANALYSIS OF ANTIMONIOUS BROMIDE.

Weight in Grammes of $Sb Br_3$ taken.	Weight of Ag Br obtained.	Per cent of Bromine.
1.2124	1.8991	66.655
0.9417	1.4749	66.647

These analyses exhibit a fair sample of the results which can easily be obtained with this method. In order to assure ourselves that the weight of the small paper disks remained constant, we have repeatedly dissolved off the small amount of adhering precipitate, and after washing and drying reweighed the disks at the completion of the analysis. Even with the larger disks there was in no case any material change in the weight, and in most cases no alteration whatever could be detected with a balance turning readily with $\frac{1}{10}$ of a milligramme.

The method of collecting precipitates here described, which, as we have shown, is so useful where considerable quantities are to be estimated, is equally applicable to very small amounts. When the quantity of the precipitate does not exceed a few milligrammes, the whole becomes fastened by the suction to the surface of the paper. There is then, of course, no need of a crucible in the process. The filter, having been dried in a watch glass, is weighed by itself, and a result of very great accuracy is reached with great rapidity. We have in this way frequently estimated minute quantities of baric sulphate and argentic chloride, whose weight proved to be only a fraction of a milligramme.

Although the apparatus here described and figured was invented independently by myself in order to overcome difficulties, already stated, which I met with in the course of my investigations, yet in its main features I was anticipated by Professor H. Carmichael, now of Bowdoin College; and I had the misfortune not to have my attention called to his paper on the subject — dated at Göttingen, 1870, and published in the Zeitschrift für Chemie, neue Folge, Band VI., 481 — until long after my own apparatus had been perfected. But although Professor Carmichael and myself started from the same fundamental idea, yet we have worked this idea out in very different forms, and with very different purposes in view. While therefore I would acknowledge most fully Professor Carmichael's priority, I have thought it best to publish this paper with the sole object of adding to his previous work the results of my own experience, and with the hope that I may thus aid in introducing into analytical laboratories what I believe to be the most important improvement in analytical chemistry which has been made since the invention of the Bunsen pump.

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